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INDUSTRIAL CROPS AND PRODUCTS

An International Journal

Industrial Crops and Products 14 (2001) 135–138

Enrichment of protein, starch, fat, and sterol ferulates from corn fiber by fine grinding and air classification☆

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Received 21 August 2000; accepted 19 December 2000

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Publication information: *Industrial Crops and Product* (ISSN 0926–6690). For 2001, volumes 13–14 are scheduled for publication. Subscription prices are available upon request from the Publisher or from the Regional Sales Office nearest you or from this journal's website (http://www.elsevier.nl/locate/indcrop). Further information is available on this journal and other Elsevier Science products through Elsevier's website: (http://www.elsevier.nl). Subscriptions are accepted on a prepaid basis only and are entered on a calendar year basis. Issues are sent by standard mail (surface within Europe, air delivery outside Europe). Priority rates are available upon request. Claims for missing issues should be made within six months of the date of dispatch.

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Abstract

Corn fiber contains hemicellulose, starch, cellulose, protein and fat. There is commercial interest to produce sterol ferulates from corn fiber oil, which accounts for only 3% of corn fiber by weight. An inexpensive process to enrich starch, protein, and fat contents of corn fiber is desirable for increased utilization and further processing. This study was conducted to determine whether fine grinding and air classification of corn fiber into separate fractions according to particle size could enrich selected components of interest. Corn fiber was finely ground in a pin mill at high speed, and the resulting ground fiber was separated into various fractions with cutpoints of 15, 18, 24 and 30 μm. The finest fraction, with a particle size of less than 15 μm, showed enriched protein, starch, fat and sterol ferulates contents compared with the starting corn fiber. The greater than 30 μm fraction was separated by sieves into five fractions. In general, protein, starch, fat and sterol ferulates contents of the fractions decreased and total dietary fiber increased with increasing particle size. Fine grinding and air classification of corn fiber can enrich protein, starch, fat and sterol ferulates contents in the fine fractions and can make further processing more economical. Published by Elsevier Science B.V.

Keywords: corn fiber; protein; starch; fat; sterol ferulates; air classification

1. Introduction

There is commercial interest to produce sterol ferulates from corn fiber oil; however, current processes are expensive because the oil content of corn fiber is only 3% by weight. An inexpensive process that can enrich valuable components in corn fiber is desirable for increased utilization.

Wet milling of corn separates the kernel into starch, germ, protein, steepwater, and fiber. Corn

☆ Names are necessary to report factually on available data; however, the USDA neither guarantees nor warrants the standard of the product, and the use of the name by USDA implies no approval of the product to the exclusion of others that may also be suitable.

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0926-6690/01/$ - see front matter. Published by Elsevier Science B.V.
PII: S0926-6690(01)00076-0

fiber contains 20–22% starch, 12–14% cellulose, 11–12% protein, 3–4% fat, and 35–40% hemicellulose (Carlson 1994, Saha et al., 1998). By treating corn fiber with enzyme, Leathers and Gupta (1996) reported that up to 70% of polymeric xylose and 100% of polymeric arabinose from corn fiber hemicellulose were liberated and most or all of the starch was hydrolyzed to glucose. Doner and Hicks (1997) isolated water-soluble hemicellulose B and water-insoluble hemicellulose A by alkaline hydrogen peroxide extraction of corn fiber. Moreau et al. (1996) extracted corn fiber to yield an oil containing sterol ferulates, which may lower serum cholesterol levels (Moreau et al., 1998). Dowd (1997) recovered approximately one-half of the starch in corn fiber by a laboratory scale wet process involving grinding, screening, washing, and centrifuging. Wet processing is usually expensive because of the need for drying and wash water disposal. Thus, we studied the effect of fine grinding and air classification on enrichment of protein, starch, fat, and sterol ferulates in corn fiber.

2. Materials and methods

Wet, de-watered corn fiber from the wet milling of corn was supplied by Williams Energy Services (formerly Pekin Energy Co.), Pekin, IL. The wet corn fiber was dried overnight at 63°C in a Proctor Dryer (Procter & Schwartz, Horsham, PA). The dried corn fiber was ground in a hammer mill equipped with a 2.36 mm diameter opening screen.

The corn fiber was ground three times at 14,000 rpm in an Alpine model 160Z pin mill (Augsburg, Germany), and fractionated in a Pillsbury laboratory model air classifier (Minneapolis, MN). The classifier was first set at a 15 μm cutpoint to obtain a fine and a coarse fraction. The coarse fraction was then classified successively with 18, 24 and 30 μm cutpoints to obtain four fine fractions (1 through 4 in increasing size) and a coarse residue (fraction 5). The >30 μm coarse residue (fraction 5) from air classification was further separated by sieving successively by sieves with square openings of 89, 124, 147, and 175 μm to obtain 30–89, 89–124, 124–147, 147–175, and >175 μm fractions. Pin milling, air classification and sieving experiments were carried out in duplicate from the same batch of oven-dried corn fiber.

Nitrogen, moisture and ash were determined by the American Association of Cereal Chemists (AACC) Methods 46-16, 44-15A and 08-16, respectively (AACC, 1995). Nitrogen was determined by Kjeldahl, moisture was determined from weight loss at 130°C for 1 h, and ash was determined gravimetrically after heating at 600°C for 2 h. Protein was calculated by nitrogen × 6.25. Starch was measured by polarimetry from a calcium chloride solution containing stannic chloride (Earle & Milner, 1944). This method is slightly different from the Corn Refiners Association (CRA) Method A-20 (CRA, 1986). Fat was measured by petroleum ether extraction in a Tecator Soxtec System 1043 extraction unit by the Association of Official Analytical Chemists (AOAC) Official Method 920.39, and total dietary fiber was determined by AOAC Official Method 985.29 (A-G) (AOAC, 1998). Composition studies were averaged from the duplicate samples.

For detecting sterols, a 0.5 g aliquot of sample was added to a 15 × 125 mm test-tube, 5 ml hexane added and extracted twice by mixing on a Vortex Genie 2 (Fisher Scientific, Pittsburgh, PA) at maximum speed for 60 min. Following each extraction, the tubes were centrifuged for 10 min at 3500 rpm to pellet the sample, and the supernatant was pipetted into a tared test-tube. Hexane was evaporated with a stream of nitrogen and gentle heat until the sample was dry, and the residue weighed.

The hexane extract was analyzed for sterol ferulates by high performance liquid chromatography (HPLC) using a diol column (5 μm, 4.6 mm × 250 mm, Lichchrosorb Diol; Alltech Associates, Inc., Deerfield, IL) and eluted with hexane:2-propanol (98:2) at a flow rate of 1.5 ml/min. The extract was dissolved in 2 ml of hexane and 20 μl was injected for each analysis. The effluent was monitored at 210 nm and 325 nm using a diode-array detector (Beckman, Model 168, Fullerton, CA). Samples were quantitated from peak heights and a standard curve was derived by linear regression using γ-oryzanol (CTC Organics, Atlanta, GA) and absorbance at 325 nm. Each sample was analyzed twice, and the results were

averaged. Injections were made by autoinjector (Beckman, model 507). Under these HPLC conditions, the sterol ferulates eluted as a group at about 9.9 min. To check for coelution of other UV-absorbing compounds, which might bias quantitation, the UV spectrum of the top of each ferulate peak was retrieved and superimposed on the peak spectrum of γ-oryzanol. Examination of the spectra showed no appreciable differences. In addition, the UV spectrum at one-third peak height before and after the maximum were retrieved and superimposed on the equivalent spectrum from the standard, and again no appreciable differences were found between the samples as compared to the γ-oryzanol standard. Duplicate samples were extracted by hexane and sterol ferulates determined by HPLC.

The data were treated by analysis of variance, and Tukey's Studentized range test was used to determine significant difference at $P < 0.05$ (SAS Institute, Cary, NC).

3. Results and discussion

The yield and composition of corn fiber and its air classified fractions after fine grinding are listed in Table 1. Fraction 1, the finest fraction with particle size less than 15 μm, had higher protein, starch, fat, and sterol ferulates, but smaller total dietary fiber content, than the starting corn fiber. Fraction 2 has double the starch, higher fat and sterol ferulates, but lower total dietary fiber content than the starting corn fiber. Fraction 3 has higher starch, fat, and sterol ferulates than the corn fiber. Fraction 5 has lower protein, starch, fat, and sterol ferulates content than corn fiber. When fraction 5 was separated by sieves into different particle sizes, the protein, starch, fat, and sterol ferulates decreased, but total dietary fiber content increased, with increasing particle size. Ash contents of all fractions in Table 1 were not significantly different ($P > 0.05$) and averaged 0.5%, and therefore were not listed individually in the table.

Because the sterol ferulates and total dietary fiber concentrations are inversely correlated, the sterol ferulates do not appear to be associated with the bulk of the fiber. Seitz (1989) dissected tissues from corn and found that the sterol ferulates were associated mostly with inner pericarp, and the aleurone was in the inner pericarp fraction. Our results seem to agree with those of Seitz (1989). Because the aleurone layer is juxtaposed to the peripheral part of the endosperm, the highest concentrations of sterol ferulates are

Table 1
Yield and composition of air classified and sieved corn fiber fractions, % dry basis

Fraction	μm	Yield	Protein	Starch	Fat	TDF[a]	Sterol ferulates	Sterol ferrulates in oil
Corn fiber			13.1 CD	15.4 CD	2.7 DE	76.4 A	0.181 E	6.7 B
1	<15	7.9[b] CD	17.1 A	23.3 B	5.5 A	41.3 C	0.306 A	5.6 C
2	15-18	10.4 CD	13.5 BCD	30.2 A	4.0 B	45.4 BC	0.210 C	5.2 D
3	18-24	30.7 B	14.3 BC	23.0 B	3.1 C	59.8 ABC	0.224 B	7.2 A
4	24-30	9.2 CD	14.8 BC	13.8 DE	2.9 CDE	62.6 ABC	0.194 D	6.7 B
5	>30	41.9 A	10.7 E	10.8 EF	1.8 F	80.6 A	0.100 G	5.5 C
5	30-89	10.7 CD	15.4 AB	19.9 BC	3.0 CD	58.1 ABC	0.112 F	3.7 F
5	89-124	12.3 C	11.7 DE	10.4 EF	2.5 E	72.2 AB	0.108 F	4.3 E
5	124-147	5.6 D	8.6 F	6.8 FG	1.9 F	79.5 A	0.074 H	3.9 F
5	147-175	6.7 CD	7.0 F	4.7 G	1.5 FG	83.0 A	0.064 I	4.3 E
5	>175	6.6 CD	6.9 F	4.0 G	1.2 G	79.4 A	0.050 J	4.1 E

[a] Total dietary fiber.

[b] Values followed by the same letter in a column are not significantly different ($P > 0.05$) from duplicate experiments. Ash contents were not significantly different ($P > 0.05$) and averaged 0.5%.

found in the fractions with the highest concentrations of starch and protein.

Moreau et al. (1996, 1999) found that both corn bran from dry milling and corn fiber from wet milling contain ferulate phytosterol esters. They indicated that corn bran oil contains up to 1.5% ferulate phytosterol esters, and that corn fiber oil may contain up to 6.75% ferulate phytosterol esters. Also, Moreau et al. (1998) observed that during dry milling most of the ferulate esters (about 80%) separate with the grits, but during wet milling they separate with the fiber. For comparison purposes, percent sterol ferulates in oil ranged from 3.7 to 7.2% for air classified and sieved fraction, and from 5.2 to 7.2% for the five air classified fractions (Table 1). No definite trend was present between particle size and percent sterol ferulates in oil. Although the 18-24 μm fraction contained higher sterol ferulates and higher percent sterol ferulates in oil ($P < 0.05$) than the whole fiber, the practical significance of this difference remains to be further evaluated. Taylor and King (2000) reported enrichment of ferulate phytosterol esters from corn fiber oil using supercritical fluid extraction and chromatography. Because air classification is relatively inexpensive, our enriched fraction(s) can be further processed by supercritical fluid extraction of ferulate phytosterol esters or by other suitable processes.

4. Conclusion

Fine grinding and air classification of corn fiber can enrich protein, starch, fat and sterol ferulates contents in the fine fractions. Selective combination of the fractions can make further processing more economical for the component(s) of interest.

Acknowledgements

The authors thank B. D. Deadmond and A. M. Kelly-Webb for technical assistance.

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